INTEROFFICE MEMORANDUM

Idaho National Laboratory INL

Date: September 13, 2013 CCN 231379

To: Paul Demkowicz, Very High Temperature Reactor (VHTR) Fuel Post Irradiation Examination Technical Lead
David Petti, Director, Very High Temperature Technology Development Office

From: Isabella J van Rooyen, VHTR Electron Microscopic and Micro-Analysis Lead

Subject: Completion of Level 2 milestone "Complete Summary Report on Advanced Microscopy Performed on Irradiated AGR-1 Specimens"

1. BACKGROUND

This memo formally documents the completion of the very high temperature reactor (VHTR) Technology Development Office (TDO) Level 2 milestone [M2GR-13IN050410], "Complete Summary report on Advanced Microscopy Performed on Irradiated AGR-1 Specimens".

The initial work scope for electron microscopy, included only basic scanning electron microscopy (SEM) examination and electron back scatter diffraction (EBSD) of selected mounted cross sectioned TRISO coated particles [1]. The basic SEM examination also included elemental analysis namely energy dispersive x-ray spectroscopy (EDS) and wavelength dispersive x-ray spectroscopy (WDS), to provide information on elemental interactions, especially at the particle layer boundaries. The basic electron microscopic examination on Advanced Gas Reactor (AGR)-1 compact 6-3-2 coated particles using SEM-EDS, SEM-WDS and transmission electron microscopy (TEM)-EDS provided some information on the micro level distribution of fission products. However this work could neither provide specific identification of silver in the fission product precipitates, nor the accurate quantification of chemical elemental compositions and distributions. This then led to the exploration of other more advanced analytical and microscopic techniques.

An overview of the entire electron microscopic examination techniques envelope planned for AGR1 particles is listed in Table 1. As analysis and interpretation of results were performed during the 2013 fiscal year, advanced techniques were expanded which is also indicated in Table 1. These expansions resulted from the knowledge gained on the electron microscopy done already as well as challenges with some of the current instrumentation or facilities. As most of these advanced microscopy and micro-analysis techniques are first- of- a- kind for irradiated tristructural isotropic (TRISO) coated particles as well as irradiated silicon carbide (SiC), the level of effort to establish facilities, path ways and methods development to perform the advanced analysis was high. This report will therefore give a summary on the maturity level of the technique development and measurements performed. In addition to AGR-1 fuel performance data; these tests have also yielded important operational experience and data on these methods and additional support activities necessary to perform this work. Detailed results will be presented in separate topical reports and/or conference or journal papers. Publications and presentations completed during the 2013 financial year on this work are also provided later in this summary report.

Table 1. An overview summary of electron microscopy and advanced micro analysis planned for AGR-1 coated particles (activities performed during 2013 are marked in green, activities not originally identified for AGR-PIE are marked with *, activities not funded but planned are indicated by "?" and activities performed ahead of plan are marked in yellow).

Technique	Compact 6-3-2	Compact 4-1-1	Compact 5-3-1 [1]	Compact 3-1-1 [2]	Unirradiated
Basic Scanning Electron Microscopy					
Basic SEM	FY2012	FY2012	FY2013	FY2013	Not planned
SEM Montage	FY2012	FY2013	FY2013	FY2013	Not planned
Advanced Microscopy Techniques					
Basic TEM	FY2012/FY2013	FY2013	Not planned	Not planned	FY1012/FY2013
EBSD [3]	FY2013	FY2013	FY2014	FY2014	FY2012
APT [4]	FY2013	FY2014	FY2014	FY2014	Not planned
EPMA[5]	Not planned	Not planned	FY2014	FY2014	FY2012
*STEM [4]	FY2013	FY2013/FY2014	FY2014	FY2014	Not planned
*EELS , EFTEM[3; 4]	FY2013	FY2013/FY2014	FY2014	FY2014	Not planned
*HRTEM[6]	FY2014?	FY2014	FY2014?	FY2014?	Not planned
*KTD[6; 7; 8]	FY2014?	FY2014	FY2014?	FY2014?	FY2014
*ASTAR[8]	FY2014?	FY2014?	FY2014?	FY2014?	FY2014?

[1] Mounted particles only delivered for electron microcopy end June 2013.
[2] Mounted particles not yet delivered for electron microcopy.
[3] Input to this method development leveraged by NSUF funded projects.
[4] CAES STEM and APT Facilities licensed for irradiated uranium containing material only from March 2013.
[5] MFC EPMA facility approved for handling of irradiated fuel September 2013.
[6] Not funded, leveraged by no-cost agreement with NMMU (South Africa).
[7] Not funded, leveraged by no-cost agreement with Bruker Nano (Germany).
[8] Not funded, submitted INL LDRD proposal, plan to do a NSUF application also (BSU, NMMU, Bruker Nano).

2. BASIC TRANSMISSION ELECTRON MICROSCOPY (TEM)

The basic TEM microscopy and analysis work completed in the 2013 fiscal year are summarized in Table 2. A brief description of selected work is provided in this summary report. Although this work could not

provide the specific detail of chemical composition of precipitates that is needed to fully understand the fission product transport mechanisms, it provided an initial indication of composition present in micron-sized precipitates. Additionally, some knowledge on the mircostructural changes due to irradiation damage was obtained, although this needs further exploration. Additionally, the samples prepared for the TEM analysis, were then used for the initial studies and feasibility tests of more advanced microscopy techniques like scanning transmission electron microscopy (STEM) and high resolution transmission electron microscopy (HRTEM).

Table 2: Basic TEM microscopy and analyses completed in the 2013 fiscal year.

Time Period	TEM Specimen	Description
Oct-Nov 2012	AGR1-632-030-1a AGR1-632-034-1a AGR1-632-034-2a AGR1-632-035-6a AGR1-632-035-6b	Analysis and interpretation on precipitates using energy dispersive spectrometry (EDS) and selected area diffraction (SAD) patterns.
November 2012	AGR1-632-035-6a AGR1-632-035-6b	Microscopy done on new focused ion beam (FIB) lamella for confirmation of Cs in SiC. Interpretation completed.
December 2012	Unirradiated particles TO-651 Variant 2 fuel TO-636 Baseline fuel	Microscopy done on SiC and Fuel kernel. Specific attention on IPyC SAD and SiC SAD for baseline interpretation with irradiated TEM microstructures. Interpretation is planned for the 2014 fiscal year.
Jan-Feb 2013	AGR1-411-030	Microscopy on 5 FIB lamellas completed. Interpretation partially completed.

2.1 Verification of Precipitate Analyses on Selected Compact 6-3-2 Samples

Although the basic TEM analysis on the compact 6-3-2 coated particles was completed in the 2012 fiscal year as described in INL/EXT-11-23911 [2], some microstructural questions remained to be addressed. Detailed analysis on precipitate was therefore completed during October to November 2012 on samples AGR1-632-030-1a, AGR1-632-034-1a, AGR1-632-034-2a, AGR1-632-035-6a and AGR1-632-035-6b. The precipitates found in the high Ag release particle, AGR1-632-030-1a, are very small with only one precipitate large enough to allow collection of selected-area diffraction patterns. Patterns from this precipitate can be indexed as zone <010>, <120>, or <131> from UPd_2Si_2 (International Centre for Diffraction Data card 00-047-1029). Because this precipitate is still small compared to the smallest selected-area aperture (effective diameter ~160 nm), most of the diffraction patterns include reflections from both the precipitate and the adjacent SiC. The presence of 2H SiC was also identified during this study. The detailed examination of the low Ag release particles, AGR1-632-034-1a, AGR1-632-034-2a, AGR1-632-035-6a and AGR1-632-035-6b, showed very similar results namely a structure similar to UPd_2Si_2. The EDS data suggests however significant compositional variations and none of the EDS analysis indicate stoichiometric UPd_2Si_2. During the interpretation and detailed analysis stages, it was decided not to continue with this work as the confirmation and quantification of the chemical composition of the precipitates will really only be verified once the advanced measurements electron energy loss spectroscopy (EELS) and atom probe tomography (APT) are completed.

Further detailed TEM examination and analysis work was also completed on the Cs precipitate observed previously in particle AGR1-632-035. This work confirmed the presence of Cs in the wormlike feature

(Figure 1). No Cs was observed in the SiC grain structure itself. The presence of Cs in the SiC structure may again only be confirmed using more advanced measurements mentioned above.

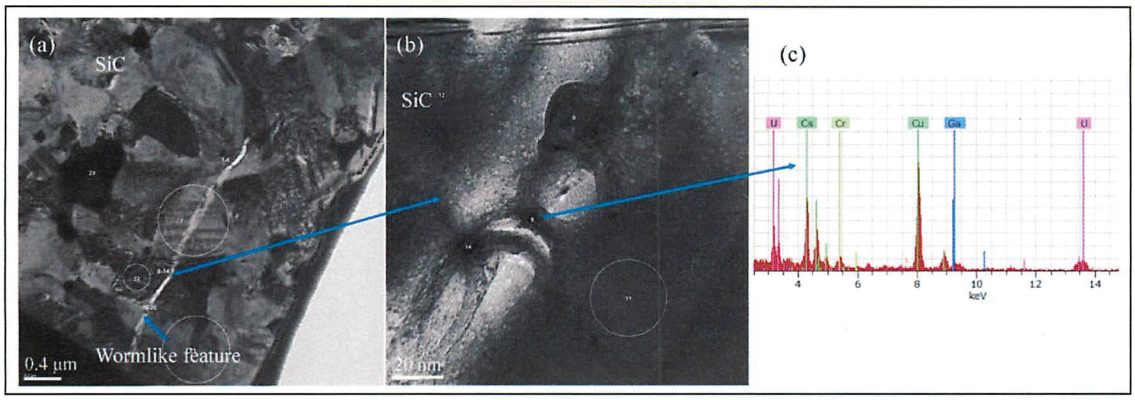

Figure 1. Image showing the Cs-containing wormlike feature in the FIB-TEM lamella AGR1-632-035 6b.

2.2 Compact 4-1-1

Although two coated particles from Compact 4-1-1, AGR1-411-021 and AGR1-411-030, were examined as part of the basic SEM examination, the original number of samples (positions) to be examined at the TEM was decreased due to equipment availability and budget constraints. This resulted in no TEM examinations for AGR1-411-021.

However, seven (7) FIB-TEM samples were prepared from AGR1-411-030 during December 2012, which included a first C-buffer sample as well. Figure 2 shows the AGR1-411-030 FIB-TEM lamellas prepared for positions 1a, 1b, 1c and 2. Subsequently the TEM examination was completed on five (5) FIB-TEM lamellas in January/February 2013. Due to limited funds, the TEM examination of the fuel lamellas was not completed, but can be if funding becomes available in the future. However, the TEM examination still provided a good basic overview on the properties of the SiC layer. The main findings are listed below:

- The TEM-EDS analysis on the micron-sized precipitates showed Pd-rich precipitates with no U and Ag detected. This is in contrast with the EDS analysis previously performed on Compact 6-3-2 micron–sized precipitates where U was identified.
- SAD measurements showed no correlation to those of UPd_2Si_2 previously found in the compact 6-3-2 specimens investigated. From both the qualitative EDS and the SAD patterns, the precipitates are different between these two compacts. This will need to be verified using more quantitative and advanced techniques.
- Slight indications of debonding at localized areas of the IPyC-SiC interface are noted. No indications of debonding were observed for the Compact 6-3-2 specimens (Figure 3).
- Cs precipitates were found in a porous region of the buffer area in specimen AGR1-411-030 (Figure 4).

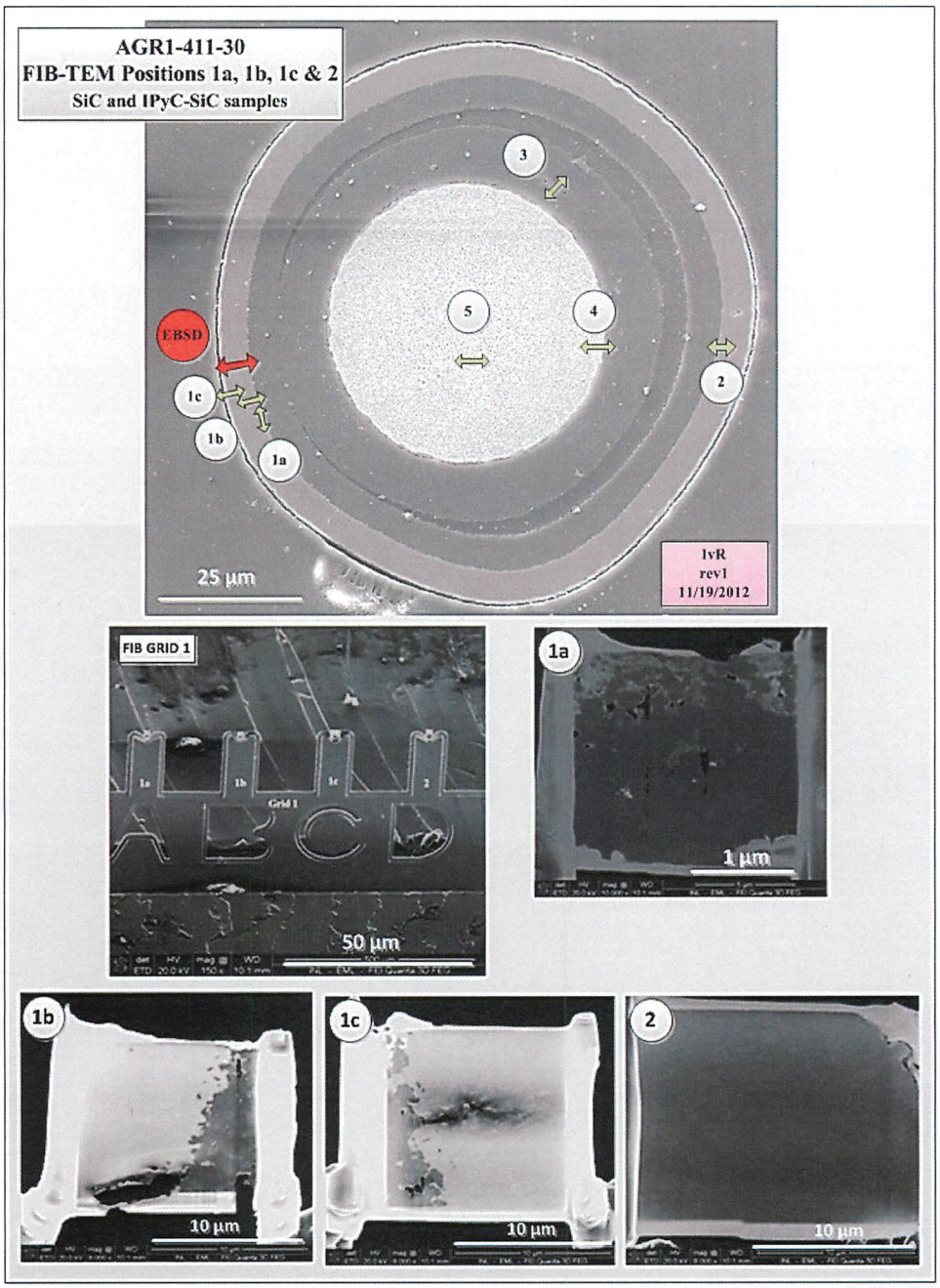

Figure 2. Image showing the AGR1-411-030 FIB-TEM lamellas prepared for positions 1a, 1b, 1c and 2.

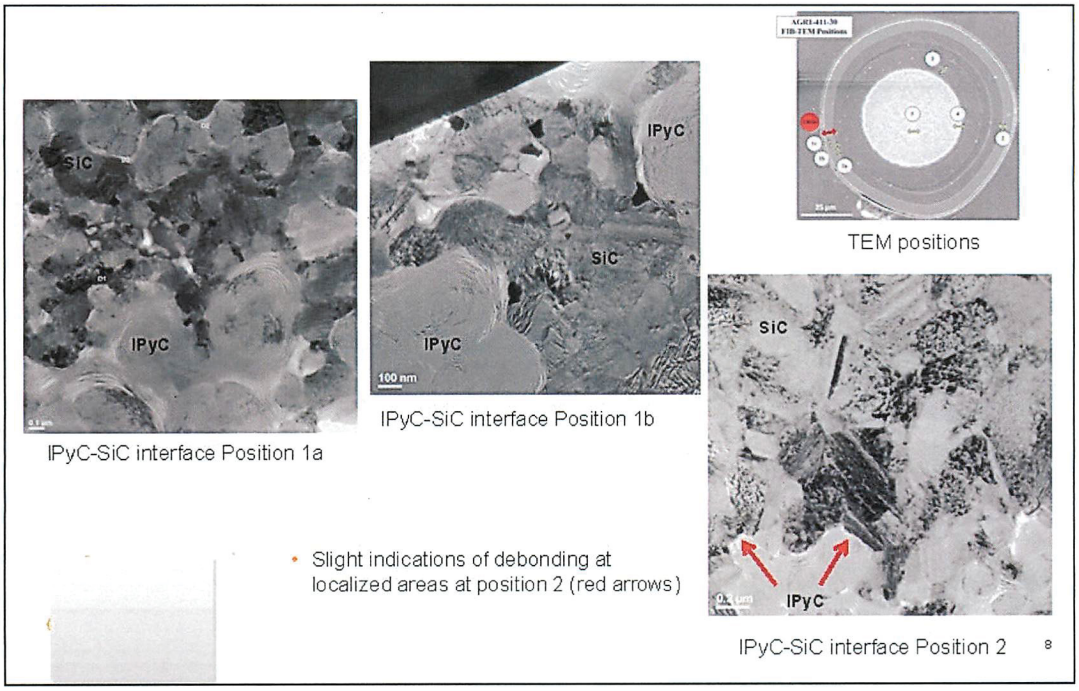

Figure 3. Indications of debonding are observed in localized areas at the IPyC-SiC interface as shown by the red arrows. Indications of debonding were not observed previously on specimens from Compact 6-3-2 [3].

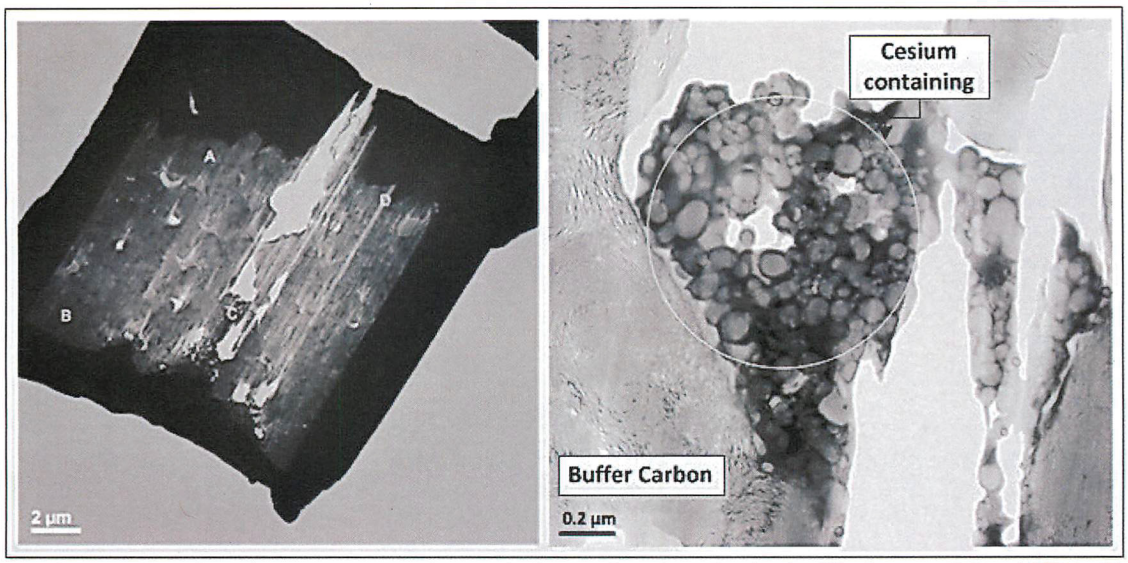

Figure 4. Image showing the presence and morphology of cesium in the buffer layer of the AGR1-411-030 particle (FIB position 3 in Figure 1) [3].

3. ADVANCED TECHNIQUES IN CENTER FOR ADVANCED ENERGY STUDIES (CAES) (STEM, EDS, EELS, ENERGY FILTERED TRANSMISSION ELECTRON MICROSCOPY (EFTEM) AND APT)

As mentioned previously, one of the biggest challenges in using these various advanced microscopy activities was to find a suitable facility that is licensed to work with irradiated materials and could meet the resolution requirements. The first approach taken was to evaluate the CAES facilities in Idaho Falls due to the advanced, high resolution electron microscopes and availability of the micro-analysis equipment. The CAES facility provided the VHTR electron microscopy team the opportunity to explore the advanced techniques of STEM, EELS, EFTEM and APT for the irradiated TRISO particles with extraordinarily significant results within the first day of analysis. The advanced microscopy work at CAES equipment (STEM, EELS, and EFTEM) completed in the 2013 fiscal year is summarized in Table 3.

Table 3. Advanced microscopy work at CAES equipment completed in the 2013 fiscal year and future work planned.

Time Period	TEM Specimen	Description
March 2013	AGR1-632-035-6b tangent samples	STEM, preliminary EELS and EFTEM Resulted in discovery of first Ag in irradiated SiC ever.
September 2013	AGR1-632-035-6b tangent samples	Post doc, NSUF funded project, more detailed EELS, EDS, and EFTEM
Started August 2013 (completion projected in Feb 2104)	AGR1-632-035-6b Radial samples	Started, shows networks through the total SiC layer
To be planned, pending funding	AGR1-632-030-1a AGR1-632-034-1a AGR1-632-034-2a AGR1-632-035-6a	Possibly NSUF funded student projects

3.1 STEM, EDS, EELS and EFTEM

The electron microscopy team used STEM, EELS and EFTEM during March 2013 to identify for the first time the physical location and elemental distribution of fission products at the micro-and nano-scale in the SiC and IPyC layers of irradiated TRISO fuel. The STEM, EELS and EFTEM analyses were conducted with a FEI Tecnai G2 F30 STEM at the Microscopy and Characterization Suite (MaCS) in CAES at INL. Figure 5 show the packaging of the first shipment of FIB lamella to CAES for further study.

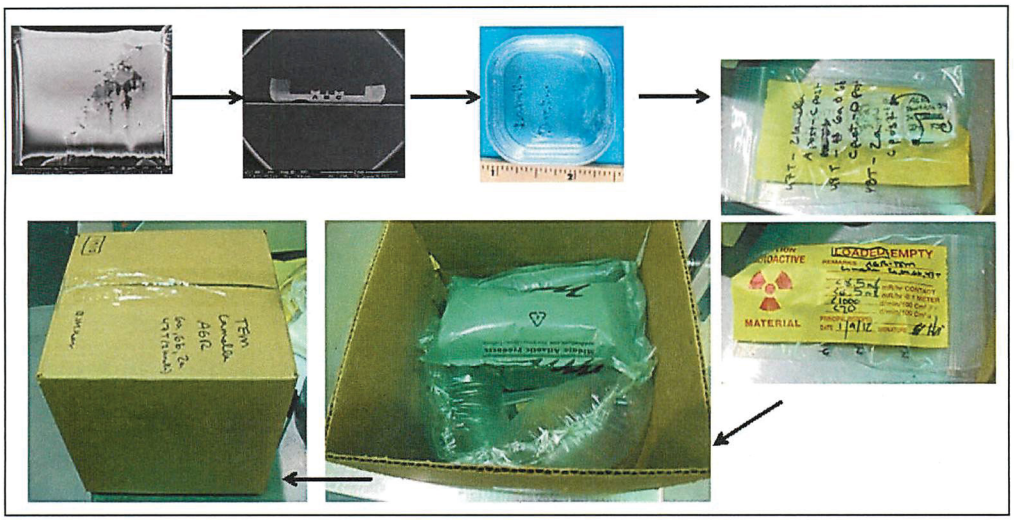

Figure 5. TEM lamella samples packaged in January 2013 for shipment to CAES for advanced microscopy in March 2013.

The first STEM specimen, AGR1-6-3-2-035-6b, was prepared as part of the original basic TEM analysis and was extracted from a location approximately tangent to the SiC-IPyC interface and contains parts of both the IPyC and SiC layers as shown in Figure 6c. This lamella was prepared at the Electron Microscopy Laboratory (EML) at the Materials and Fuels Complex (MFC) of INL using the dual-beam Quanta 3D FEG FIB. No additional FIB lamella was used during this initial and highly successful STEM examination.

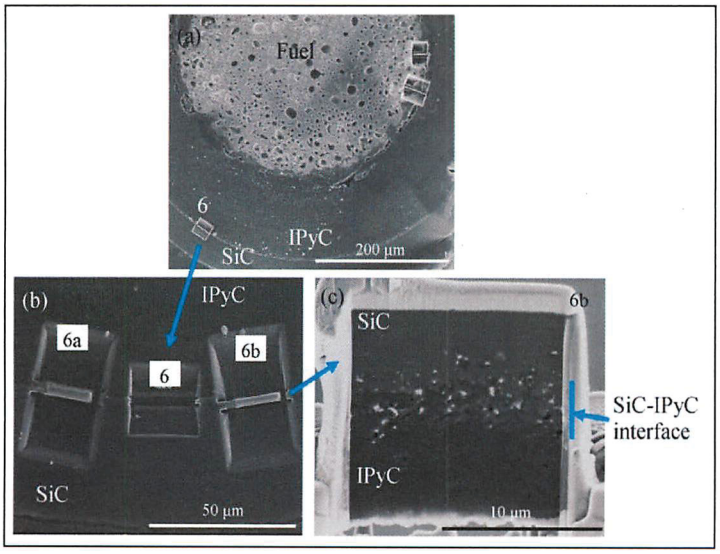

Figure 6. Images showing: (a) the cross sectioned mounted coated particle AGR1-632-035, (b) the FIB lamella position 6b; and (c) the AGR1-632-035-6b FIB lamella consisting of the SiC-IPyC interface at higher magnification.

The STEM examination provided evidence of nano-sized silver precipitates at triple-points (Figure 7) and grain boundaries in the SiC on the edge of the SiC-IPyC interface. Cadmium was also found to be present in the triple junctions (Figure 8).

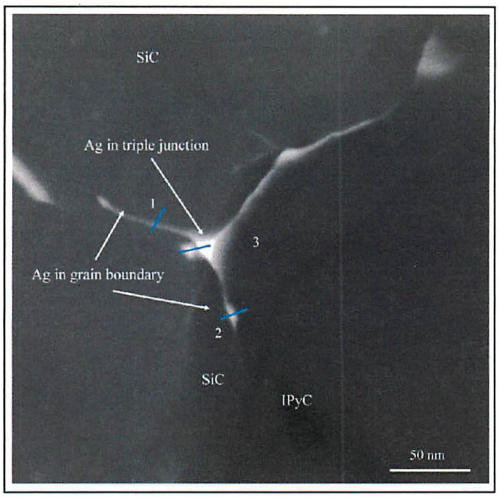

Figure 7. Image showing the high angle annular dark field (HAADF) STEM image of silver-containing grain boundaries and triple junction at the outermost edge of the SiC adjacent to the IPyC [4].

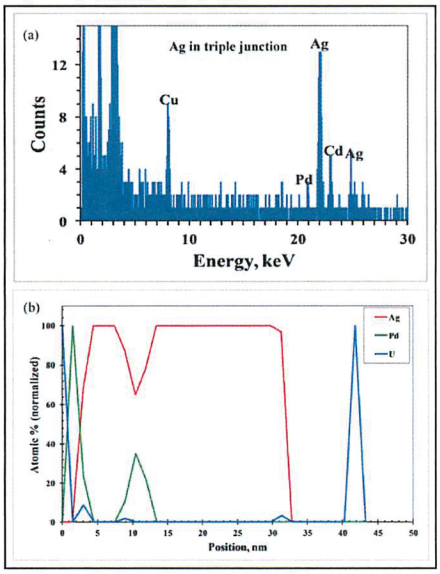

Figure 8. Image showing (a) the EDS spectrum identifying the Ag K peak at 22.162 keV and the presence of Cd in the centre of the triple junction shown in Figure 7, and (b) the EDS line scan profile through the triple junction indicating the presence of Ag. Although no Pd is observed in the triple junction, Pd and U are identified in the SiC matrix adjacent to this triple junction [4].

Palladium was identified as the main constituent of the micron-sized precipitates present at the SiC grain boundaries which confirmed the preliminary studies by Van Rooyen *et al* [2]. Additionally spherical nano-sized palladium-rich precipitates were found inside the SiC grains (Figure 9). No silver was found in the center of the micron-sized fission product precipitates using these techniques, although silver was found on the outer edge of one of the Pd-U-Si containing precipitates which was facing the IPyC layer. Only Pd-U containing precipitates were identified in the IPyC layer and no silver was identified in the IPyC layer.

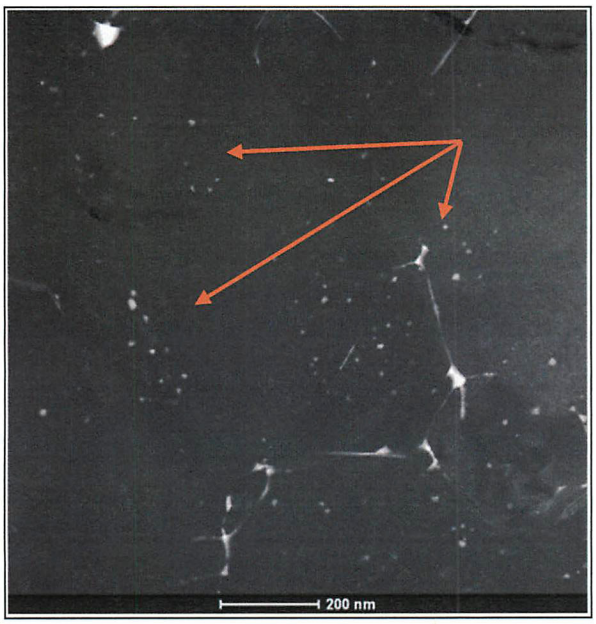

Figure 9. Palladium-rich precipitates inside the SiC grains and at the SiC grain boundaries in a particle from irradiated AGR-1 Compact 6-3-2 [4].

A fine network of the Pd/Ag containing material is visible on grain boundaries inside the SiC layer, although this needs to be explored further. It was found in the earlier studies [3] that the micron-sized Pd-rich precipitates were visible up to approximately 15 μm from the SiC/IPyC interface.

It was decided in June 2013 to expand this initial STEM examination with lamella prepared in the radial direction. These radial samples will provide through-thickness information on the grain boundary nano-networks of fission products, which will be important in the mechanistic transport studies. FIB preparation of these radial-positioned specimens from AGR1-632-035 and the STEM began in August 2013 and will continue until February 2014. The aim of this work on radial samples is to determine the:

- Depth of fission product networks into the SiC layer. Although just starting with the examination, it is important to note that evidence was found of nano networks of fission product transporting through to the outer edge of the SiC (Figure 10).
- Depth of silver and Pd radially in the SiC. Initial work has started on selected triple points only, and although Ag and Pd were found in conjunction with each other, no observation can be made with regards to the depth of Ag found in these networks.

- Concentration profile of fission products as a function of radial distance away from the SiC-IPyC interface. Preliminary EDS line-scan measurements show chemical composition variations through the same triple point. This will be further examined.

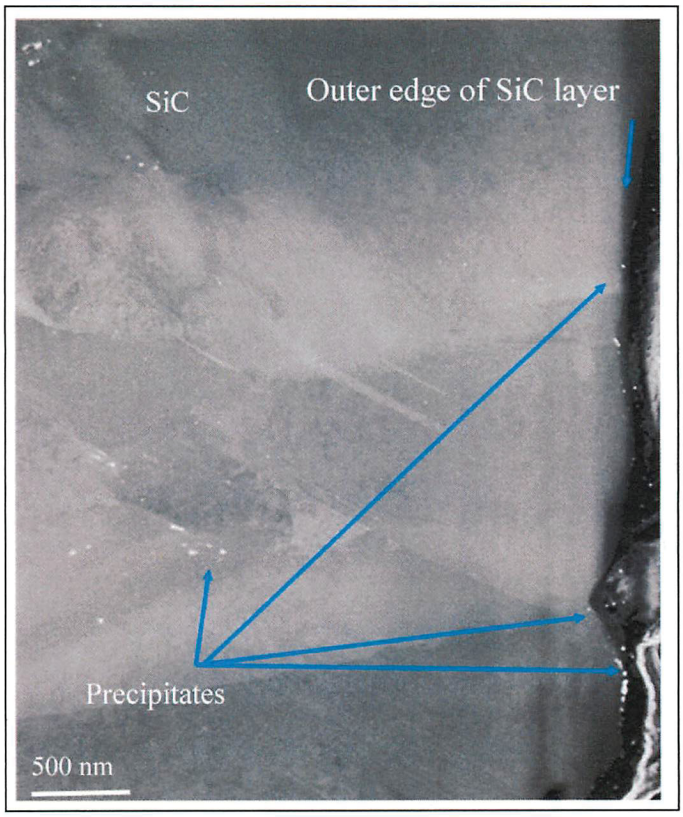

Figure 10. STEM micrograph showing evidence of nano network of fission product transporting through to the outer edge of the SiC.

In our strategy to identify silver, another analytical tool, EELS is considered. In TEM, some incoming electrons lose energy when they travel through the specimen due to the inelastic interaction with the specimen's atoms. EELS/EFTEM requires thin TEM specimens. EELS analyzes the energy distribution of these scattered electrons and provides quantitative compositional information about the nature of the atoms under illumination by the electron beam. The transmitted electrons can be filtered with respect to energy loss and only those electrons with a specific energy loss, (e.g., that associated with Ag) can be chosen for imaging. In essence, these filtered electrons, with a selected energy loss, form an elemental map in the imaging mode (EFTEM). EELS analysis is considered important for TRISO fuel research because of the specific resolution of elements of interest. In EELS analysis, the chemical sensitivity and the size of resolvable feature could be at 1% and smaller than 1 nm, respectively. Moreover, Pd, Ag and U have close but separable edge energies in an EELS spectrum, which suggests that trace amounts of Ag in the studied sample should be able to be detected using the EELS technique. EELS/EFTEM requires thin TEM specimens.

Ag, Pd and U can be distinguished by using EELS since their edge energies are separated from each other (i.e., Pd M4,5 335 eV, Ag M4,5 367 eV, U N7 381 eV)[5]. Therefore, EELS was carried out at the triple point (Figure 7) where an Ag-rich intergranular phase was found by EDS.

Figure 11a is a zero-loss image of the triple-junction area and the triple junction phase is in darker contrast in this image. A corresponding EELS spectrum in a range from ~ 230 eV to ~ 390 eV within this area is shown in Figure 11b. It can clearly be seen from this spectrum that the Ag M-peak (387 eV) is detected, providing direct evidence of an Ag-rich phase along the grain boundaries. A very small peak at 340 eV is also observed in the spectrum, which likely corresponds to a Pd M-peak, indicating this area may contain some small amount of Pd. EFTEM elemental maps of Ag and Si were also obtained at this triple-junction area, as shown in Figures 11c and 11d, respectively. In the EFTEM elemental map, the lighter region corresponds to the elements being analyzed. Therefore, Figure 11c clearly shows the triple-junction phase is Ag-rich; and Figure 11d indicates this triple-junction area is located at the boundary between SiC and IPyC phases.

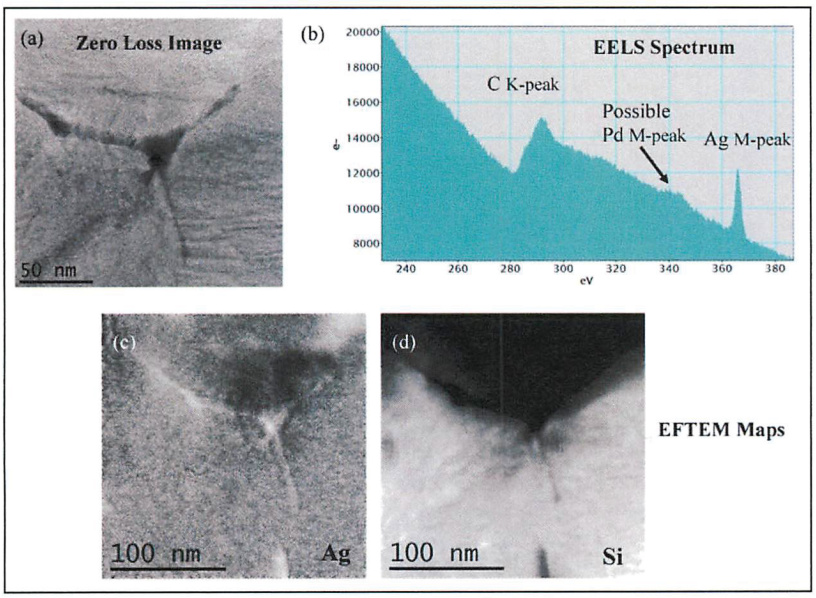

Figure 11. Zero-loss image of the second phase at triple junction (a) and corresponding EELS spectrum obtained at this area (b); EFTEM elemental maps of Ag and Si at this area (c) and (d), respectively [4].

Further work on EELS and EFTEM will continue under an NSUF funded project in FY-2014. This NSUF rapid prototype experiment funding request was awarded in March 2013 in collaboration with Wisconsin-Madison University. As part of that work, the research will further expand the level of detail on the TEM-EELS and APT work to supplement the VHTR funding. The initial APT measurements have started. The proposed study aims to gain insight into the transport mechanisms of fission products (FP) released from neutron irradiated TRISO fuel through compositional analysis of FP precipitates and FP grain boundary (GB) segregation in the silicon carbide (SiC) layer of the TRISO fuel.

3.2 Atom Probe Tomograpy (APT)

The first APT probe tips were prepared using the FIB at EML from specimen AGR1-632-035 during July 2013. Particle AGR1-632-035 was chosen for this first APT analysis as Ag was identified in the SiC structure during the earlier STEM examination in March 2013. Two tip sections were chosen for the APT analysis, one tangent and one radial to the SiC-IPyC interface. In total 15 tips were fabricated (Figure 12). To date, three (3) APT analyses involving five (5) tips were completed on the first APT sample set as shown in Table 4.

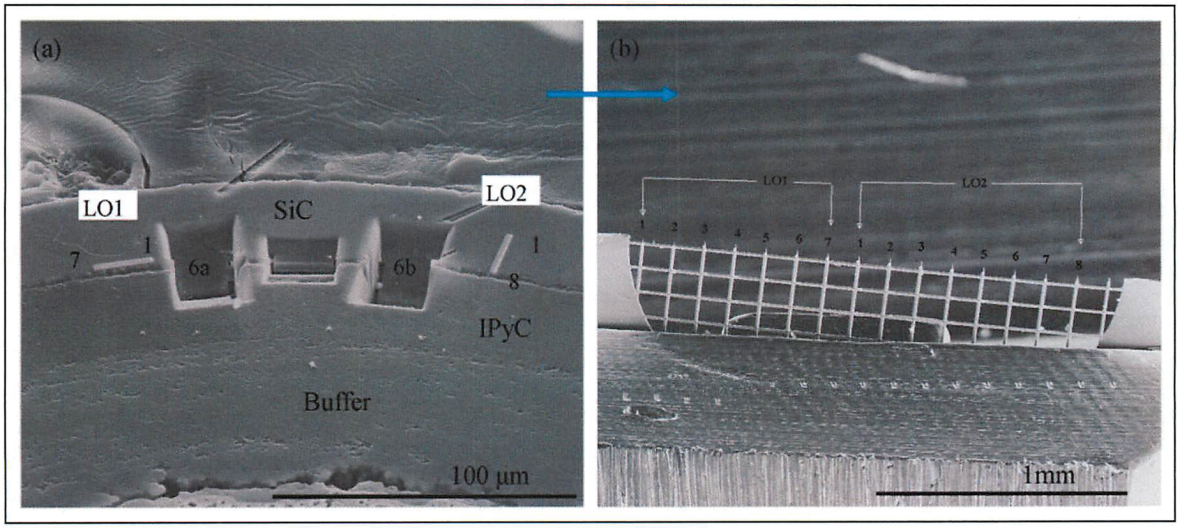

Figure 12. The locations of the two tip sections are shown in (a) with the placement of the respective tips on the copper grid holder in (b).

Table 4. APT analyses completed in the 2013 fiscal year on particle AGR1-632-035.

Date of APT Analysis	APT Tip Analyzed
7/24 TEM examination of APT samples	All 15 tips
7/25-7/26 (2 days)	LO1-7 LO2-8
8/6-8/7 (2 days)	LO2-3 LO2-5 LO2-6
Planned for 9/9-9/10 (2 days)	LO2-2

These first-of-a-kind APT analyses on irradiated SiC provided numerous valuable lessons and actions were identified to resolve or minimize these challenges in future. During our first APT run, we could not obtain useful data due to frequently tip fractures. By complimentary TEM characterization, a surface oxidization layer of about two nanometers thick was identified on top of the SiC phase at the top of sample tips. We believe this oxidation layer also contributes to the tip fracture. The biggest challenges experienced during the first APT preparation and APT analysis are summarized as follows:

- The copper grid was too flexible therefore causing premature fracture of the first tips analyzed
- The oxide layer on the tip contributed to premature fracturing of tips. FIB fabrication caused Ga deposition on the outer layer.
- The location of fission product features, specifically the nano-sized Ag rich triple points as these nano-sized features are not detectable by the FIB-SEM and therefore not possible to fabricate the APT tip with these nano-sized features in the top part of the tip.
- Fractures of the tips could also be due to the inherent properties of irradiated SiC. At present no APT measurements have been completed on irradiated SiC anywhere else in the world.

In the latest APT run on August 6-7 2013, the first irradiated SiC was successfully analyzed up to a depth of 37.6 nm as shown in Figures 13 and 14. Significant progress was achieved in obtaining useful data within the SiC phase by altering running parameters, i.e., sample temperature (50 ~ 80 K) and laser energy (100 ~ 200 pJ), to overcome tip fracture. As a result, data from a volume size of 36.6 nm × 35.7 nm × 37.6 nm containing about 10.9 million ions was obtained from the TRISO sample. However, the running parameters are a lot higher than usual, which may introduce artificial or even false information that cause difficulties in data reconstruction and analysis. Therefore, the next step will be to focus on optimizing running parameters to obtain large enough data with minimal artificial/false information to accurately represent the structure of the SiC layer in the irradiated TRISO coated particle. It is envisioned that optimization of the laser parameters can be established with the remaining sample, LO2-2 planned for October 2013.

Various actions resulting from the lessons learned during the analysis of the first sample set, will be incorporated in the second round of APT samples planned for October 2013. The objective with this second round of APT tips is to increase the depth of APT analysis in the top 200nm of the tip closer to the typically optimal value for analysis. Focused attention will also be drawn to the fabrication parameters as well as the location of tips in close proximity of the micron –sized fission product precipitates. This is a function of experiment design and the skill of the operator, which although not easy should improve with experience.

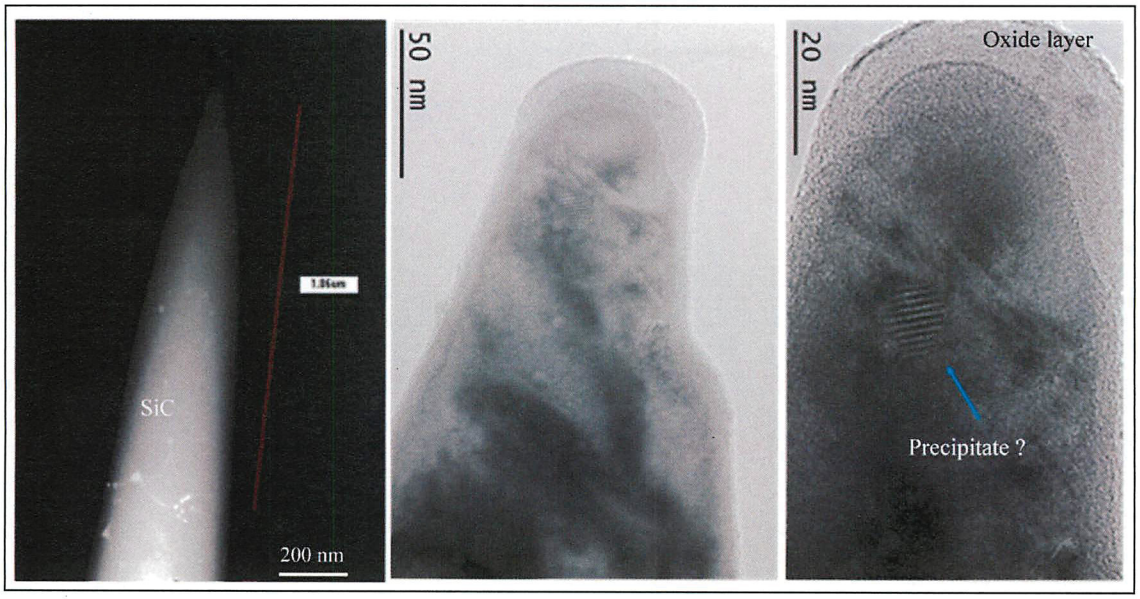

Figure 13. Images of tip LO2 tip-5 showing the tip profile and microstructural features at higher magnification from left to right. The STEM image (left) shows the different locations of the nano-grain boundary networks and nano precipitates.

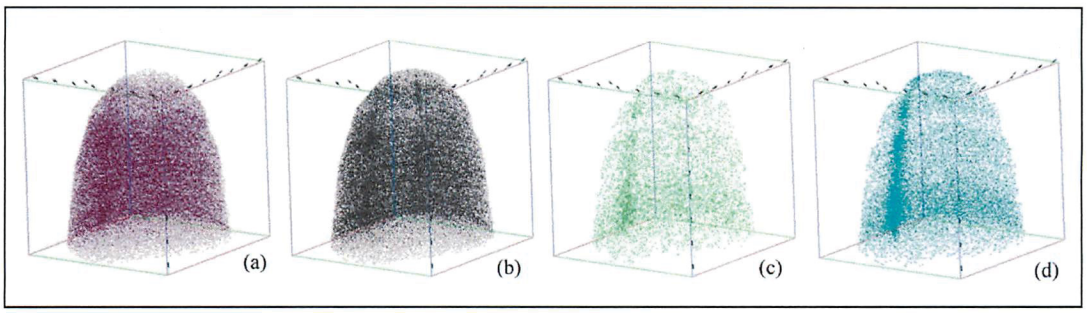

Figure 14. Example of selected chemical element distribution in the top part (38 nm) of the LO2-5 tip. This shows the presence of the (a) C; (b) Si; (c) He or H and (d) O_2.

4. HIGH RESOLUTION TRANSMISSION ELECTRON MICROSCOPY (HRTEM)

HRTEM examination was performed at the Nelson Mandela Metropolitan University (NMMU), South Africa on a focused ion beam prepared specimen AGR1-411-030 positions 1b and 2. Both of these FIB specimens were cut radially to the SiC-IPyC interface as shown in Figure 2. The FIB preparation took place at EML at MFC using the standard process for preparing TEM samples.

The microscope used for the study was a JEOL ARM 200F TEM operated at 200 kV accelerating voltage in scanning transmission mode. The microscope is equipped with two CEOS spherical aberration correctors for correction in TEM and STEM modes, as well as an Oxford Instruments XMAX 80 EDS detector and Gatan Quantum Image filter with dual EELS capabilities. Imaging and analysis of the sample were done using a sub-angstrom sized probe with a probe current density of approximately 68 pA. It was determined during analysis that this probe gave the best balance of resolution, count rate and sample sensitivity. For compositional analysis, the main technique used was EDS. This was due to the relative accessibility of the higher energy K edges for Ag and Pd that could be used for independent identification of their presence due to the sufficient separation of the position of the edges in energy. Another distinct advantage of the microscope used was that of the simultaneous acquisition of both bright field and dark field STEM images. This aided in the identification of grain boundaries in conjunction with elemental atomic number contrast.

The main objective of this examination was to obtain very high resolution images at atomic level, of the fission products. Three days were dedicated to this examination with extraordinary results. Atomic resolution images were obtained showing Pd and Ag atoms co-existing on the same location at the SiC grain boundaries (Figure 15 and 16). In contrast with the STEM results obtained from AGR1-632-35b to date, it was found that Ag and Pd always co-exist in the same triple point. This is first-of-a-kind results and this new data has a high impact potential on the Ag transport mechanism. Additionally more detail is obtained on the intra-and intergranular movement of Pd in the SiC layer (Figure 17). These results will be presented in a journal article during the next couple of months. The HRTEM examination was done in collaboration with Prof. Jan Neethling and Dr. Jaco Olivier. It was extremely beneficial to be present during this examination, as Dr. van Rooyen directed examination as results became available and direct knowledge was gained on these images during the collaborative discussions.

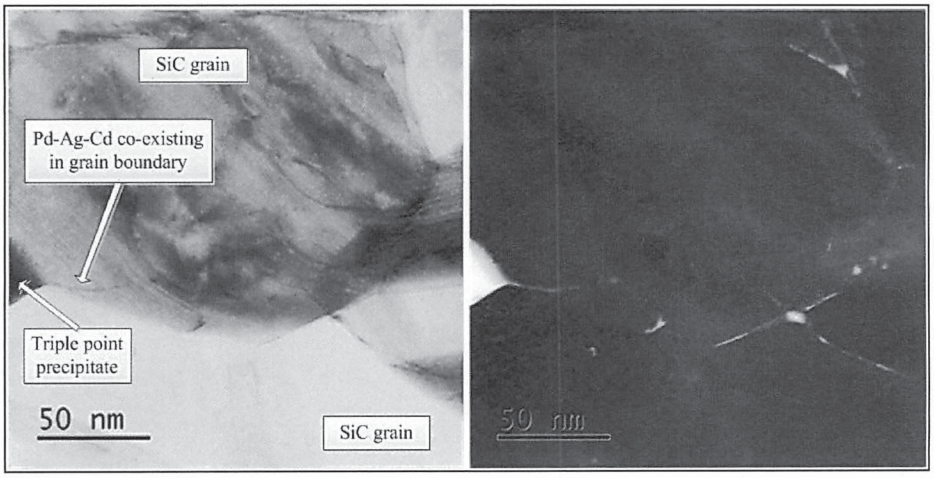

Figure 15. Bright field (left) and dark field (right) STEM images of fission product networks on grain boundaries and triple points of specimen AGR-141-030 position 1b. Pd-Ag-Cd was found co-existing at location indicated by arrow (see Figure 16 for higher magnification images of this area).

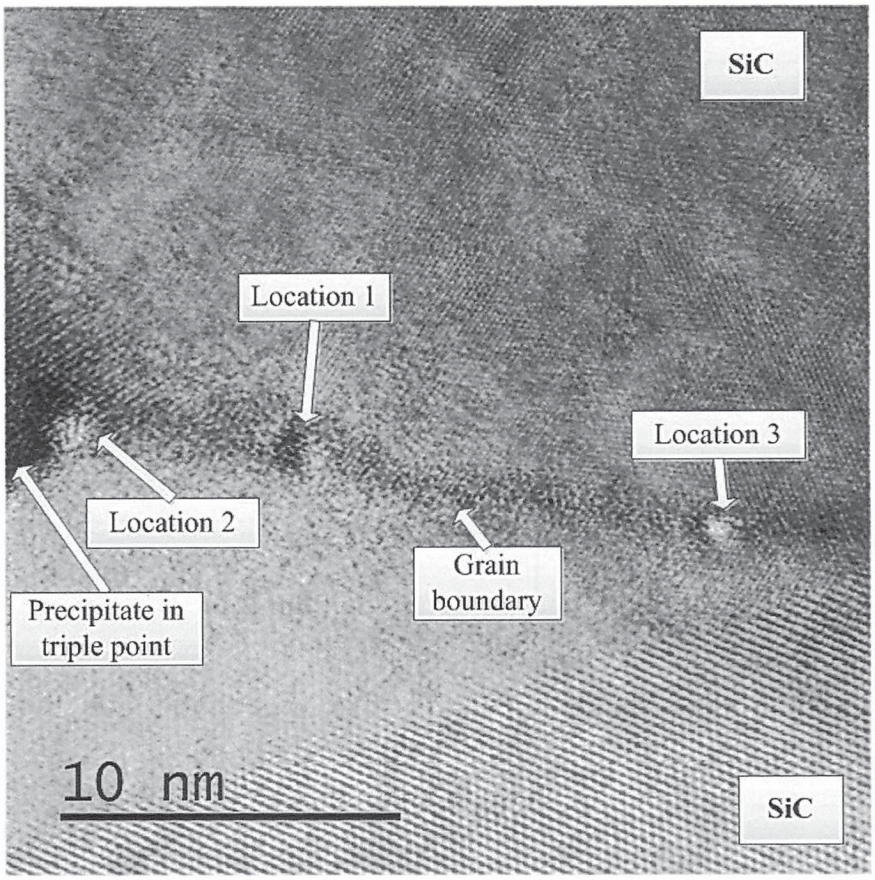

Figure 16. High resolution bright field image of co-existing Pd-Ag-Cd atoms at location 1 on a grain boundary between two SiC grains of specimen AGR-141-030 position 1b. The EDS analysis on locations 2 and 3 shows only Pd using the EDS analytical tool. The black dots on the grain boundary and at the

triple points are the respective Pd, Ag or Cd atoms. From this image it is not possible to differentiate between those atoms of these elements because they all show up as "black". The EDS analysis caused the slight damage to the atomic structure as shown by the spherical feature at the location arrow.

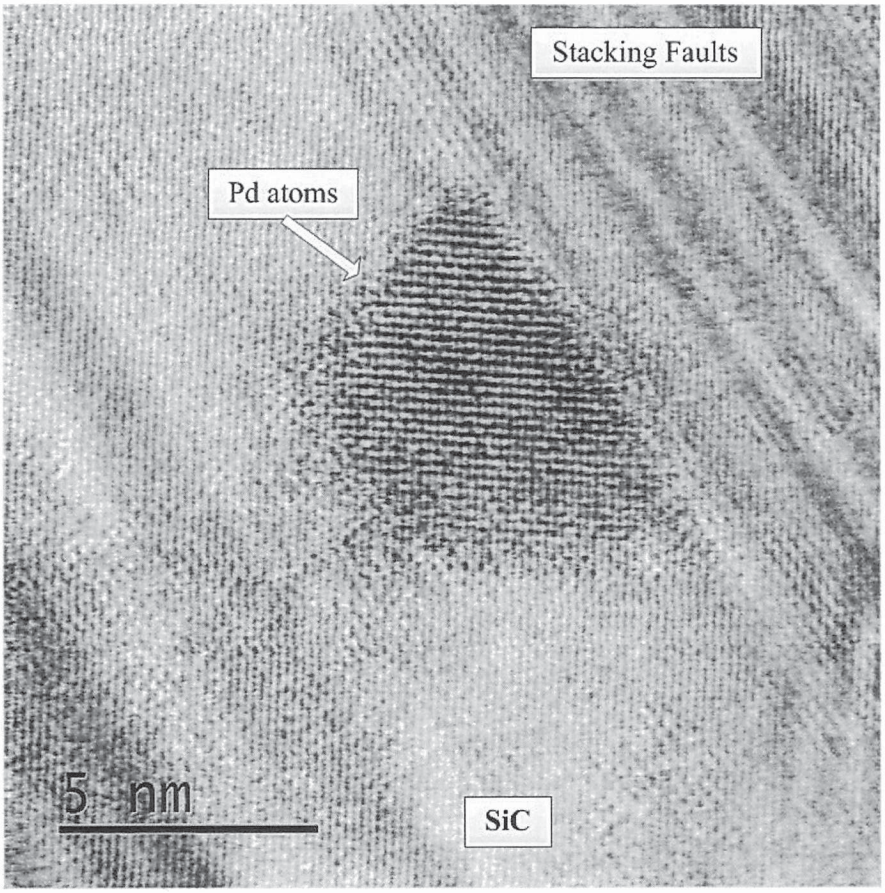

Figure 17. Bright Field high resolution TEM image of a Pd precipitate (black dots are the Pd atoms) agglomerated at a stacking fault in the SiC structure.

5. TECHNIQUES TO DETERMINE CRYSTALLOGRAPHIC INFORMATION (EBSD, TKD, ASTAR)

EBSD is a practical characterization technique to obtain crystallographic information like crystal type, orientation, grain boundary characteristics, grain size distribution and texture. These measurements are obtained from small areas using a SEM. The ability of this technique to perform scanning over a wide range of step sizes makes it possible to investigate the microstructure down to nano levels when needed [8]. Data from this type of analysis provides details of the high angle grain boundary distribution to test recent grain boundary-Ag transport mechanism hypothesized by researchers [9-11].

EBSD has been the dominant method for obtaining crystallographic data within the SEM for decades. It is applicable to most crystalline materials except for ultrafine grained materials with grain/cell diameters smaller than ~100 nm. This limitation is based on EBSD technique's spatial resolution, which is a function of the backscattering coefficient of the analyzed material, the electron probe diameter and energy, and the incident angle between the beam and the specimen surface (~20°). While significant

spatial resolution improvements can be achieved by decreasing the beam acceleration voltage and diameter there is still a multitude of nanomaterials that are very difficult or impossible to characterize using the EBSD technique.

SiC is a difficult material to image with EBSD without any crystallographic disruption due to irradiation damage, lattice strains and multiple polytypes of SiC present. With Si and C both being fairly light elements, the signal generated for EBSD collection is weak. The unirriadiated SiC materials that were examined previously possess fairly well preserved microstructures and are therefore measureable. However, as a crystal structure is stressed mechanically, by heat treatment or by neutron exposure, the Kikuchi patterns (lines) will become weak and as the material approaches an amorphous nature, the lines may not be existent at all. This behavior provides an additional challenge for the collection of EBSD data on irradiated SiC layers.

Transmission Kikuchi Diffraction (TKD) or t-EBSD is an SEM method for measuring crystallographic properties in materials, similar to EBSD, but with an order of magnitude improvement in spatial resolution. The resolution is better because of the forward-direction scattering that excites the electrons near the exit surface, where they have a chance for Kikuchi scattering. Forward scattering is of course favored in conventional EBSD as well, but in order to form a pattern, backscatter detection out of the beam entry surface is also required, and the backscatter signal is less compared to the transmission case. Another advantage of the forward-scatter TKD geometry is that because low-angle elastic scattering is favored over high-angle scattering, there is less beam-spreading in thin specimens by the time the important Kikuchi events occur. In addition, the interaction volume is considerably smaller.

TKD has been successfully applied to studies of nanoparticles, thin films and thinned foils obtained from bulk materials. Thus, all known TEM sample preparation methods will work, and the INL preferred way is FIB preparation of TEM lamella. Incident beam energies in the 15 keV to 30 keV range can be used, along with probe currents in the range of a couple hundred picoamperes to a few nanoamperes. Dwell times for both point and mapping modes are typical of those used for reflection EBSD.

ASTAR from NanoMEGAS is a new technique in TEM, which fully utilizes the high spatial resolution potential of TEM (e.g., < 2 nm with FEG TEM) to do crystal orientation and phase mapping on nanometer-size grains-automatically. The ASTAR was recently installed on the TEM (FEI Tecnai F30 STEM FEG) at CAES. Unlike the EBSD-SEM which relies on Kikuchi lines obtained from the sample surface, in the EBSD-TEM mapping, controlled by the NanoMEGAS DigiSTAR unit, the electron beam is scanned across the sample area of interest. Then electron diffraction (spot) patterns are obtained and the individual patterns compared with pre-calculated templates via cross-correlation matching techniques for orientation and phase mapping. Particularly, combined with electron beam precession diffraction mode, the technique provides ultra-precise orientation and phase maps. The orientation resolution is < 1nm. The precession mode improves electron diffraction quality by increasing the number of diffraction spots (almost double) and avoiding Kikuchi lines, which is also the only possible way to distinguish multiple phases in one sample. The ASTAR technique also provides an ultra-fast data acquisition with external/internal charge-coupled device (CCD), e.g., < 5 min for an area of 5 × 5 μm^2, 20 nm step size. Thus, ASTAR will be capable of providing both crystallographic orientation and phase mapping, providing grain boundary/precipitation orientation relationships, grain boundary character identification and dislocation sub-structure. These are valuable pieces of information in the understanding of atomic processes during irradiation.

Figure 18 shows a schematic overview of the work done as part of AGR-1 PIE with complementary projects and other funding sources. Although the initial focus started to obtain crystallographic information from conventional EBSD patterns, technical difficulties and operational hold-ups caused a

wider thinking process. The progress on EBSD and TKD measurements will be briefly discussed in the following sections. The detail of samples analyzed to date is provided in Table 5.

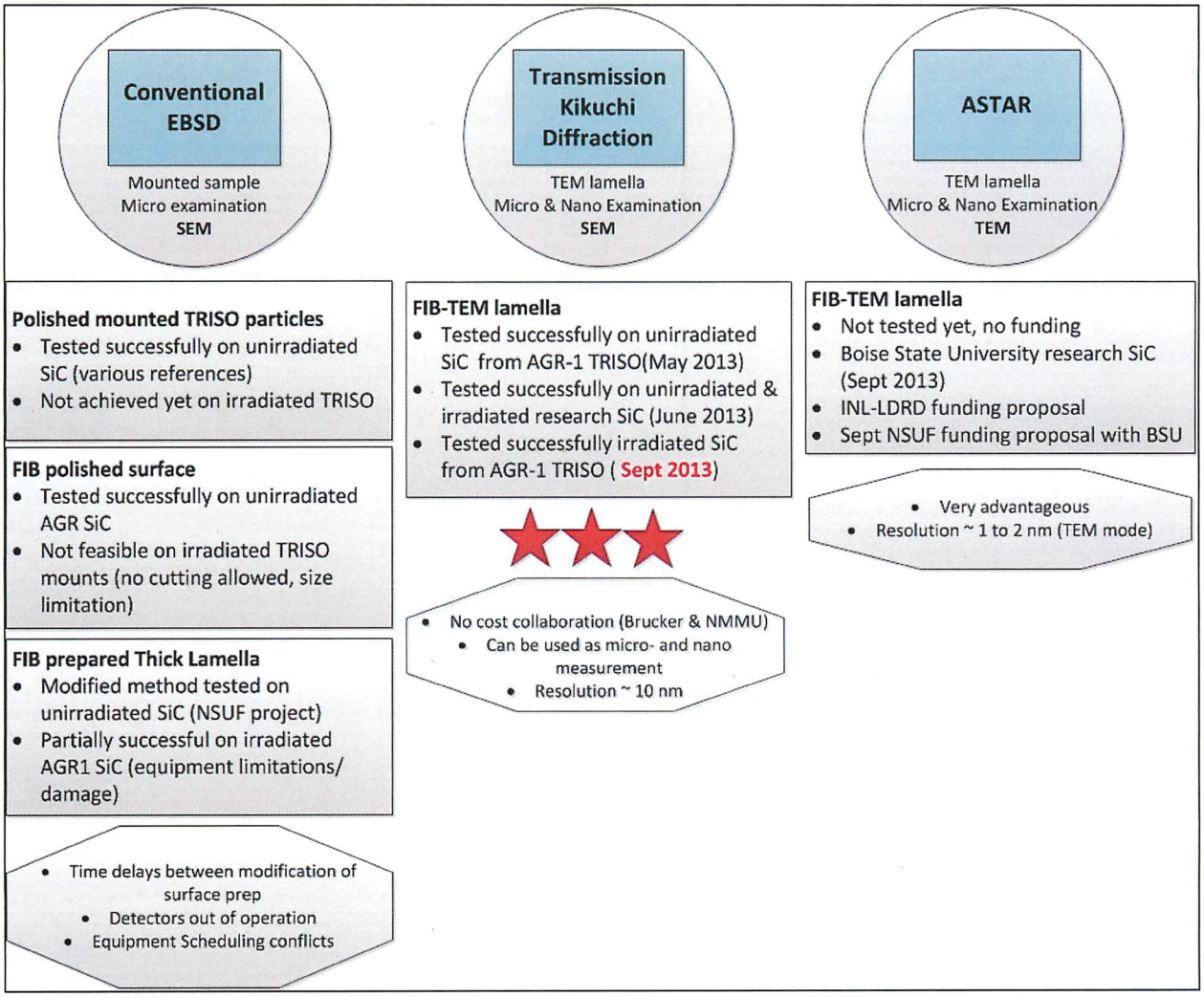

Figure 18. Schematic overview of the work done as part of AGR-1 PIE with complimentary projects and other funding sources (stars indicating the most successful results obtained at present).

Table 5. Crystallographic analyses completed in the 2013 fiscal year.

Date	Sample Analyzed	Technique Used
Oct 2012-April 2013	FIB prepared AGR1-632-034 AGR1-632-035 AGR1-411-030	EBSD in EML-FIB; only partial results could be obtained on AGR1-632-034 Delays due to software and hardware malfunction
May/June 2013	FIB prepared AGR1-632-034 AGR1-632-035 AGR1-411-030	EBSD in EML-FIB: In spite of FIB-repolishing, recalibration of detector, re-aligning detector, verifying the positioning of samples, better results could be obtained, although grains can be clearly seen on the SEM image itself.
August 2013	Conventional polished prepared AGR1-531-031 AGR1-531-038	EBSD in EML-JEOL7000 Delayed as EBSD software was disconnected
Feb 2013-May 2013	FIB-TEM lamella Unirradiated SiC TO-651-Sample 6, which represents the Variant 2 AGR1	TKD (Brucker) (no cost collaboration) FIB lamella already prepared for basic TEM, no extra cost for PIE; Successful results, feasible to proceed with optimization and irradiated samples
Sept 2013	FIB-TEM lamella AGR1-411-030-1b AGR1-411-030-1c AGR1-411-030-c	TKD (NMMU) (no cost collaboration) Successfully obtain measurements, still need to be further optimized, analyzed and interpreted. This may replace the immediate need for conventional EBSD.

5.1 Electron Back Scatter Diffraction (EBSD)

Although EBSD measurements on the unirradiated SiC layer of TRISO coated particles have been reported by various researchers [12 – 14], no such EBSD measurements have been reported on irradiated SiC. Sample preparation quality is one of the largest challenges with small grained material and specifically with the differential removal rates of IPyC and SiC of the TRISO coated particles. The latter

easily results in a rounding effect of the SiC at the SiC-IPyC interface which causes the EBSD analysis to be partially ineffective for fission product transport studies.

Preliminary comparative work on sample preparation techniques was reported on unirradiated SiC [10]. This study by Van Rooyen et al. [13] described a new approach to the FIB preparation technique used previously by Kirchofer et al. [8]. In this modified FIB sample preparation technique, a thicker TEM lamella was prepared as shown in Figure 19. The initial TEM-lamella-version of this technique resulted in vertically mounting the sample but a more recent modification resulted in horizontal mounting. Although no EBSD data has been obtained yet from the horizontal mounting technique, it provides additional advantages when working with irradiated materials. It is expected that no extra handling will be necessary and standard grid holders can be used. This will substantially decrease the risk of exposure to the microscopist and the risk of losing a radioactive sample. As long collection times seem to be necessary for SiC, drifting may cause a loss of data. The modified horizontal mounting substantially decreases the possibility of drifting during lengthy data collection.

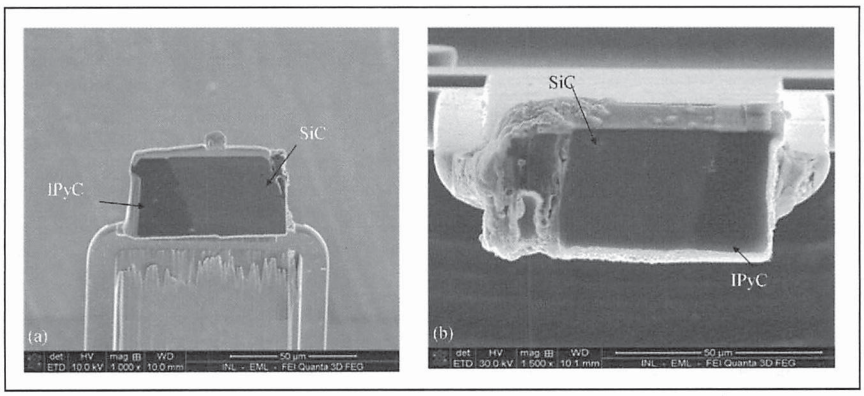

Figure 19. Micrographs showing a) vertical and b) horizontal FIB prepared thick EBSD lamellas on irradiated SiC layers of TRISO coated particles of AGR1-632-034 and AGR1-141-030 respectively.

Data collection was acquired from a FIB prepared sample of AGR-1-632-034 and the inverse pole figure (IPF) orientation map collected showing no preferred orientation (Figure 20). It is clear from Figure 20 that further improvements are necessary, but these initial images provide a baseline baseline for our work going forward. Clear grains are visible in secondary electron SEM images so current EBSD hardware limitations (e.g., camera) for this type of material are also being evaluated. It is hard to tell if the lack of well-defined EBSD patterns is an artifact of the detector type, SEM operating parameters, sample preparation, or material damage from irradiation. A disadvantage to using the FIB-SEM system for EBSD collection, is that no back scatter images could be taken to assist in the identification of precipitate location.

To date, no EBSD data collection using the EML SEM Jeol700 could be done on the as–polished cross sectioned particle (AGR1-531-038 (mount 20U) due to operational issues. It is established that the location of the particle in the mount, will be sufficient for EBSD data collection (Figure 21(a)). Additionally, it is seen from the topographical image in Figure 21(b) that the SiC grains are nearly visible in the back scatter mode. This is promising for EBSD data collection; although still needs to be proven.

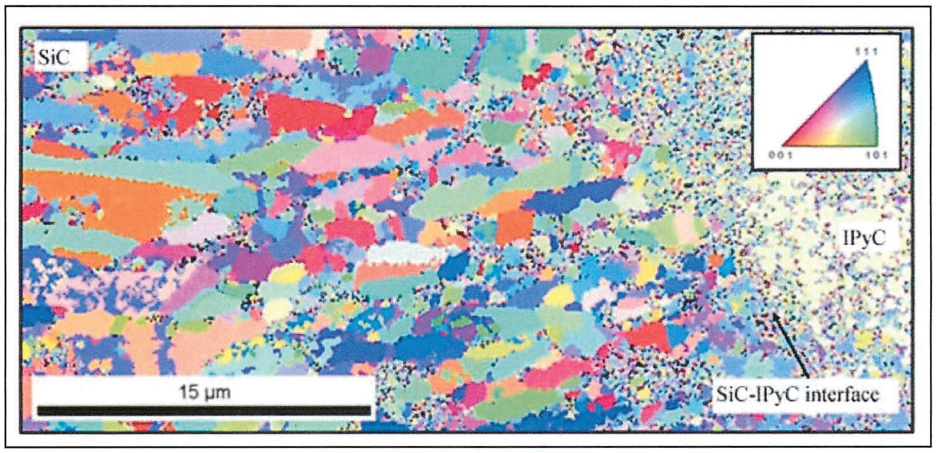

Figure 20. Inverse pole figure (IPF) orientation map collected from a neutron irradiated SiC layer from sample AGR1-632-034 showing no preferred orientation [14].

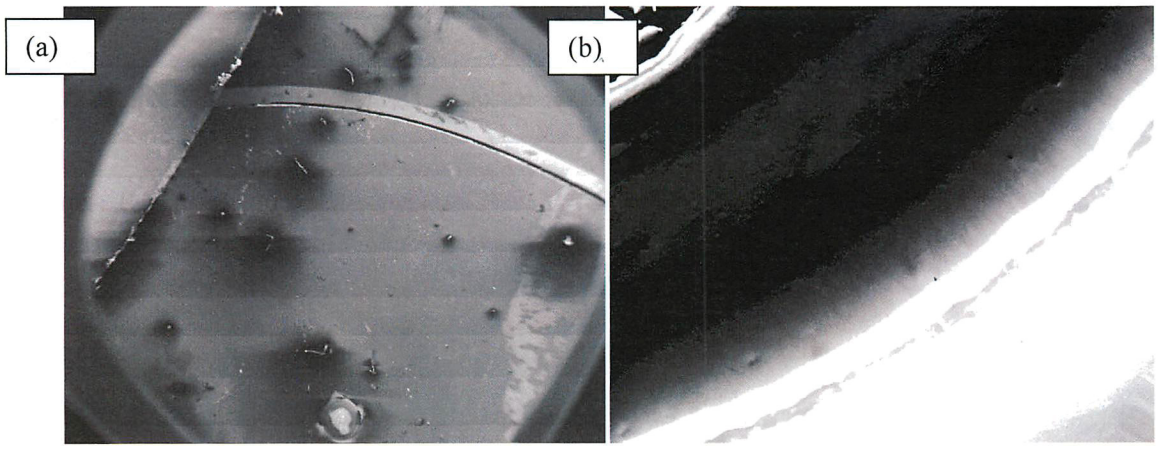

Figure 21. Images showing (a) the AGR1-531-038 particle approximately 3 mm from the edge in mount 20U which should be an adequate placement to get the EBSD camera close to get good diffracted electron density, and (b) topo graphic image using the two sector BSE detector in "TOPO" mode to show subtle relief features. This image already shows some indications of grains.

5.2 Transmission-EBSD (t-EBSD) (also called Transmission Kikuchi Diffraction [TKD])

For initial exploratory work, t-EBSD measurements were performed on a non-irradiated FIB-prepared sample, TO-651-Sample 6. This represents the Variant 2 AGR-1 fuel fabricated with SiC deposition parameters similar to the Baseline fuel with changes only to the IPyC coating deposition parameters producing a less dense IPyC layer. The FIB–prepared sample was fabricated from the SiC layer in the radial direction just inside the SiC layer. The advantage of using a sample from this batch material is that EBSD results are available from work performed by Kirchofer et al. [8] and can be used for comparison, although differences in area of examination need to be accounted for. In this report only preliminary results are provided from the first t-EBSD measurements and comparative evaluation will be provided at a later stage after full optimization of the t-EBSD method. Once this technique has been successfully demonstrated on unirradiated SiC, it will be applied to the irradiated SiC. The orientation map (IPF) overlapped with the pattern quality map (PQM) is shown in Figure 22. No preferred orientation is

observed in this area. Figure 23 below shows the distribution of CSL boundaries (Σ3 and Σ27a) in blue and green respectively. The map was acquired using a step size of 8 nm. The results indicate that a high fraction of the boundaries are Σ3 boundaries. This information should only be considered qualitative as quantitative results will require less zero solutions near the grain boundaries. More measurements with improved spatial resolution will be required for a quantitative grain boundary analysis.

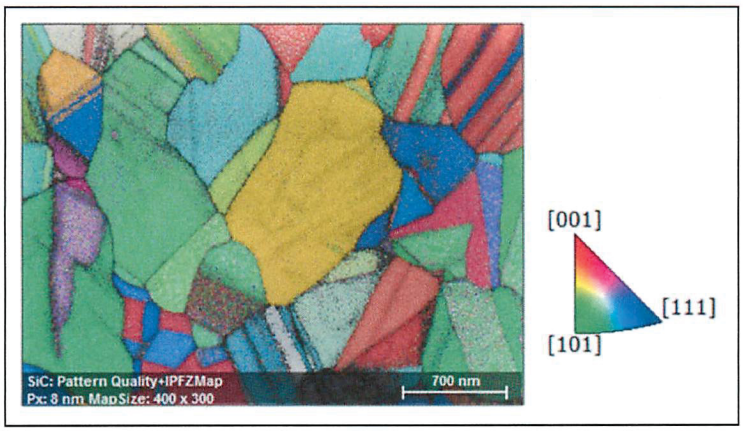

Figure 22. Inverse pole figure orientation map, (IPF), overlapped with Pattern Quality map (PQM) [14].

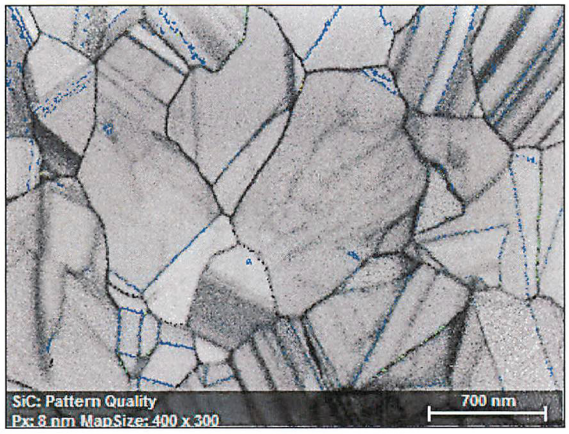

Figure 23. PQM and grain boundaries map showing special Σ3 and Σ27a boundaries in blue and green respectively while all other grain boundaries are shown in black [14].

Future collaborative work at NMMU includes the measurement of the grain boundary characteristics using t-EBSD (TKD) on various samples. Although this work was only recently initiated, successful TKD measurements were already obtained. Detailed analysis still needs to be completed but an example of the inverse pole figure obtained on AGR1-411-030-2 is shown in Figure 24. This may provide validation for the work that is envisioned to be completed at INL as part of the future EBSD work on advanced microscopy at CAES and IRC.

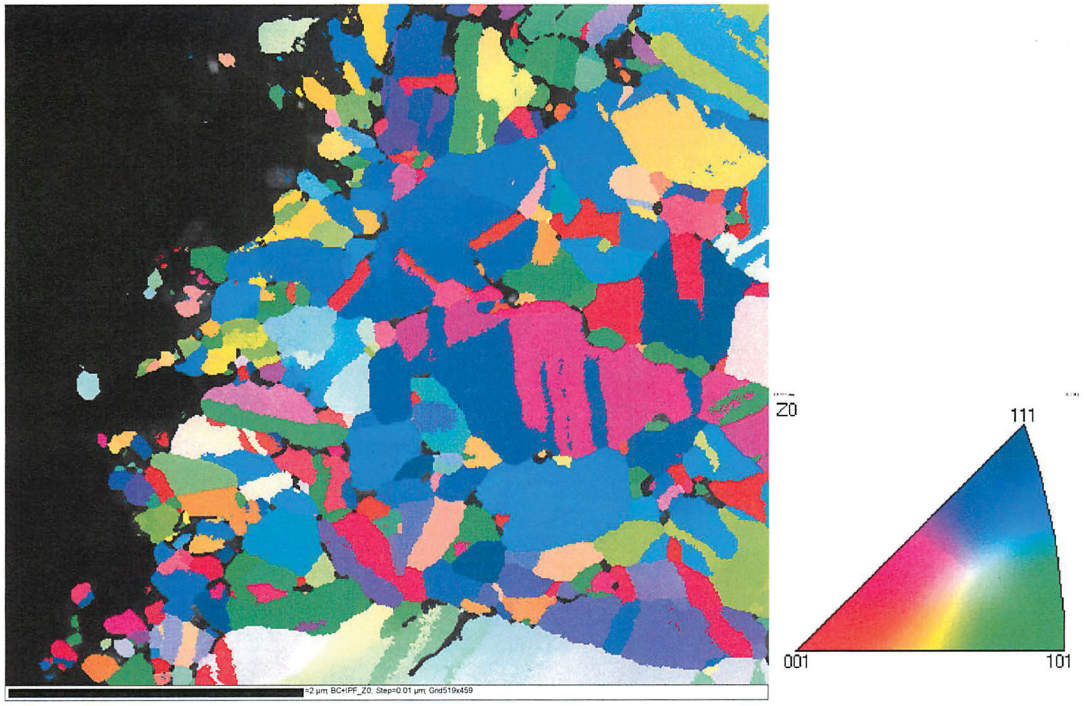

Figure 24. Inverse pole figure (IPF) orientation map collected from a neutron irradiated SiC layer from sample AGR1-411-030-2.

6. ELECTRON PROBE MICRO-ANALYZER (EPMA)

Although the EPMA measurements are scheduled for the 2014 fiscal year mainly because the facility was not approved for the handling of irradiated fuel until early in September 2013, some preparatory work was already completed. This included completing the necessary laboratory instructions, radiological safeguards and ALARA reviews for the work. A mock-up using a sample blank was completed to demonstrate the safety and efficacy of irradiated sample handling procedures. Additionally it was necessary to change the sample mounts from 1 ¼ inch to 1 inch to fit in the EPMA sample holder. Because of the EPMA sample holder's tight tolerances, it was necessary to use a chemical decontamination procedure on actual mounted irradiated TRISO coated particle samples rather than a contamination fixation procedure. Recent attempts to load the sample into the EPMA show that the modified mount does not fit in the EPMA sample holder, as it is approximately 0.007 inches too large for the holder. It is now being determined whether the sample holder can be modified to accommodate the current mounted samples. These findings will be monitored and investigated to ensure that critical dimensional tolerances are conveyed to the mounting of the samples.

7. MATURITY LEVEL OF TECHNIQUES AND METHOD DEVELOPMENT

Significant progress was made on enhancing the knowledge of the performance of TRISO coated particles after neutron irradiation using advanced microscopic analysis. As can be seen from the previous sections in this summary report, some techniques and measurements are more developed than others. The information provided in Table 6 provides a snapshot of the maturity level at this time.

Table 6. An overview summary of the maturity level of the electron microscopy and advanced micro analysis techniques and facility readiness for irradiated materials at this time.

Technique	Facility Readiness		Technique Maturity	
	NRC Licensed	Approved Admin Controls	Equipment	Parameters Optimization
Basic TEM	Yes MFC-EML	Yes	Good	Good
EBSD	Yes MFC-EML	Yes	Low slow speed camera (FIB) unknown method and software (JEOL7000) operation and maintenance	Low low confidence index on preliminary patterns
	Yes CAES	No decided not to expose equipment to irradiation	Good NSUF funded work on unirradiated SiC successfully completed	Good NSUF funded work on unirradiated SiC successfully completed
	Yes IRC	Not yet Expected readiness November 2013	Good NSUF: unirradiated SiC successfully completed	Good NSUF: unirradiated SiC successfully completed
APT	Yes CAES	Yes	Good	Medium Good progress from 1st runs
EPMA	Yes MFC-AL	Yes Sept 2013	Good	Low 1st run to start
STEM	Yes CAES	Yes	Good	Good
EELS EFTEM	Yes CAES	Yes	Good	Low Only 1 run completed

Technique	Facility Readiness		Technique Maturity	
	NRC Licensed	Approved Admin Controls	Equipment	Parameters Optimization
**HRTEM	Yes NMMU	Yes	Good	Good
**TKD	To be confirmed Brucker Germany	To be confirmed	Good	Medium 1sr run on unirradiated SiC very successful
	Yes NMMU	Yes	Good	Medium 1st run on irradiated SiC very good results
ASTAR	Yes CAES	Yes	Medium Partly shared with BSU in Boise, hardware needs to be transferred when required	Low Initial work on unirradiated SiC in progress at BSU, no irradiated SiC done yet
** No equipment available at INL or close to INL at this moment				

8. PUBLICATIONS, PRESENTATIONS AND FUNDING APPLICATIONS

Several publications and presentations were drafted or completed in the past fiscal year on advanced microscopy as part of the AGR-1 PIE work package. Publications funded from other sources, although complementary and used as input to this work, are not listed below:

- Completed full paper to be presented at the Global 2013 conference. "Advanced electron microscopic techniques applied to the characterization of irradiation effects and fission product identification of irradiated TRISO coated particles from the AGR-1 experiment," IJ van Rooyen, YQ Wu, TM Lillo, TL Trowbridge, JM Madden and D Goran, 29 September 2013.
- Presented: "Electron Microscopic Characterization of AGR-1 TRISO Coated Particles", IJ van Rooyen, NMMU, Physics department, 19 July 2013, Port Elizabeth, South Africa.
- Journal of Nuclear Materials paper was accepted in September 2013 for publication: "Identification of Silver and Palladium in Irradiated TRISO Coated Particles of the AGR-1 Experiment," I. J. van Rooyen, T. M. Lillo and Y.Q. Wu.
- Presented at the INL NS&T annual peer review meeting, "Identification of Silver in Irradiated TRISO Coated Particles of the AGR-1 Experiment Using Advanced Electron Microscopic Techniques," IJ van Rooyen, 5 June 2013.

- Submitted paper for INL Nuclear Fuels and Materials Spotlight volume 4,: "First-of –a-kind advanced electron microscopy characterization applied to identify silver and palladium-containing fission products in irradiated TRISO coated particles of the AGR-1 experiment," I. J. van Rooyen, T. M. Lillo and Y.Q. Wu.
- Presented at HTR2012 conference. "Electron Microscopic Evaluation and Fission Product Identification of Irradiated TRISO Coated Particles from the AGR-1 Experiment: A Preliminary Review", Paper HTR2012-3-023, Proceedings of the HTR 2012, Tokyo, Japan, October 28 – November 1, 2012. IJ van Rooyen, DE Janney, BD Miller, PA Demkowicz, J. Riesterer.
- Presented at HTR2012 conference. "Silver (Ag) Transport Mechanisms in TRISO Coated Particles, A Critical Review," Paper HTR2012-3-040, Proceedings of the HTR 2012, Tokyo, Japan, October 28 – November 1, 2012. IJ van Rooyen, ML Dunzik-Gougar, and PM van Rooyen.
- Completed SEM section of Compact 4-1-1 AGR PIE milestone report during December 2012
- Issue updated Compact 6-3-2 Electron microscopic report (INL/EXT-11-23911) December 2012, "Electron Microscopic Examination of Irradiated TRISO Coated Particles of Compact 6-3-2 of AGR-1 Experiment," Isabella J van Rooyen, Brandon Miller, Dawn Janney, Jessica Riesterer, Paul Demkowicz, Jason Harp, Scott A Ploger".
- Paper submitted to Journal of Nuclear Design (through HTR2012 organizers) in January 2013. "Silver (Ag) Transport Mechanisms in TRISO Coated Particles, A Critical Review," IJ van Rooyen, ML Dunzik-Gougar, and PM van Rooyen.
- Submitted to Journal of Nuclear Design (through HTR2012 organizers) in January 2013. "Electron Microscopic Evaluation and Fission Product Identification of Irradiated TRISO Coated Particles from the AGR-1 Experiment: A Preliminary Review," IJ van Rooyen, DE Janney, BD Miller, PA Demkowicz, J. Riesterer.

9. CONCLUSIONS

Significant success was achieved during this time period and can be summarized as follows:

- The STEM, TEM, HRTEM, EBSD, TKD and EELS results are the first of kind results on irradiated SiC layer of TRISO coated particles reported in open literature.
- The STEM examination provided the first-of-a-kind results indicating silver containing nano-sized triple-points and grain boundaries. This provides significant knowledge for silver transport mechanistic studies which has been the topic of international research for the past forty years.
- The HRTEM analysis provided the first image to the atomic level of Ag and Pd atoms on the grain boundaries of irradiated SiC. This also provided insight into the co-existing nature of Pd and Ag in the same grain boundary.
- Additionally, the recent TKD results obtained on the irradiated SiC, are the first of such results at INL on any fuel or material. This is also the first such results internationally in the VHTR community on actual irradiated particles.

Although all the advance techniques discussed in this report are not fully optimized yet, the results obtained in such a short period of time are providing useful information to the VHTR program.

10. ACKNOWLEDGEMENTS

The experimental work and data analysis presented here have been performed by the INL VHTR fuels electron microscopy team and collaborators:

- Scott Ploger and Jason Harp: Mount and decontamination preparation for electron microscopy examination and micro-analysis
- Dawn Janney: Basic TEM microscopy
- Jim Madden: FIB-TEM and APT sample preparation; EBSD sample preparation improvement
- Tammy Trowbridge: EBSD data collection and input to sample preparation techniques
- Thomas O'Holleran: Preliminary EBSD microscopy on Jeol7000 SEM
- Yaqiao Wu (Boise State University): APT, STEM, FTEM and EELS microscopy
- Tom Lillo: Basic TEM, EBSD, APT, STEM, FTEM and EELS microscopy
- Bin Lin (University of Wisconsin-Madison): Contribution towards APT measurements
- Daniel Goran (Brucker-Nano): TKD of unirradiated SiC
- Jan Neethling, Mike Lee, Jaco Olivier, William Goossen (Nelson Mandela Metropolitan University): TKD and HRTEM of irradiated SiC
- Karen Wright: EPMA
- Isabella van Rooyen: VHTR fuels electron microscopy and micro-analysis lead

The contributions of staff at several key MFC facilities (Hot Fuels Examination Facility, Analytical Laboratory, and Electron Microscopy Laboratory) and CAES in completing this work are also gratefully acknowledged.

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